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AN EVALUATION OF PROCESSES FOR THE BLACKENING OF PRECIPITATION-HARDENED STAINLESS STEELS

JOSEPH T. MENKE

JUNE 1979

TECHNICAL REPORT

U.S. ARMY ARRCOM

ENGINEERING DIRECTORATE

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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Precipitation-hardened stainless steels (corrosion-resistant materials with excellent mechanical properties) require dark, nonreflective finishes to be useful for most military applications. Work involving such finishes was conducted to determine which of the various blackening processes could be used for these materials without decreasing their inherent corrosion resistance.

Corrosion tests were conducted on coated and uncoated 17-4, 17-7, 14-8Mo, and AM350 alloys in the annealed and precipitation-hardened condition. The black-

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ening processes evaluated included black chromium plating, zinc phosphating, molten sodium dichromate, and alkaline-oxidizing treatments. Activated and passivated specimens were used as controls in the corrosion tests. Potentiostatic, potentiodynamic, and 5% salt spray (ASTM-B117) tests were used to evaluate the various specimens.

Salt-spray tests showed that maximum corrosion protection was provided by black chromium and zinc phosphate coatings. All corrosion tests showed that black coatings produced by the alkaline-oxidizing process provided varying degrees of corrosion resistance, dependent upon the alloy or thermal treatment used, and that coatings produced by the molten sodium dichromate treatment provided poor corrosion protection. Black coatings on the 14-8Mo and AM350 alloys exhibited the best corrosion resistance.

FOREWORD

This work was authorized as part of the Manufacturing Methods and Technology Program of the U.S. Army Materiel Development and Readiness Command, and was administered by the U.S. Army Industrial Base Engineering Activity.

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INTRODUCTION

Precipitation-hardened (PH) stainless steels are used in many military applications because these materials offer high strength, corrosion resistance, and fabricability. Tensile strengths, often in excess of 200 KSI, are obtained by solution heat-treating and precipitation hardening. Because these metallic surfaces are usually bright, such alloys used in military hardware must be blackened to produce a nonreflective surface. Work involving such surface treatment was conducted to select the best process for the blackening of PH stainless steels.

The specified blackening treatment for stainless steels is that of the molten sodium or potassium dichromate bath process. However, these types of treatments have been shown to warp certain hardware configurations and to reduce the inherent corrosion resistance. Therefore, alternative treatments (alkaline-oxidizing, black chromium plating, and zinc phosphating) were evaluated for this project.

PROCEDURE

The precipitation-hardening stainless steel alloys, e.g., 17-4 PH and 17-7 PH frequently used for components of small arms weapons, were selected for investigation. Specimens of each alloy were cut from a single sheet of solution-annealed material. The compositions of the alloys tested are presented in Table 1.

TABLE 1

Composition of the Stainless Materials Tested (Weight Percent)

Alloy\Element	Carbon	Chromium	Nickel	Manganese	Other
17-4	0.02	16.0	4.4	0.26	3.3 Cu
17-7	0.08	17.1	7.4	0.60	1.16 Al
14-8	0.03	15.0	8.4	0.00	2.3 Mo; 1.14 Al
AM350	0.09	16.1	4.3	0.64	2.8 Mo
17-4(Casting)	0.26	15.5	4.2	0.14	3.3 Cu

Specimens were evaluated in the solution-annealed and the precipitation-hardened conditions. The thermal treatments to accomplish precipitation hardening of the various alloys are summarized in Table 2.

The test specimens were 2" x 3" x 0.03" panels that were blasted with 250-mesh glass beads. Precipitation-hardened specimens were descaled in a 10% nitric-2% hydrofluoric acid solution before glass-bead blasting. Annealed and precipitation-hardened specimens were subjected to the following blackening treatments after glass-bead blasting:

- Molten dichromate: MIL-C-13924 Class 3 "Black Oxide Coating for Ferrous Metals".
- Alkaline-oxidizing: MIL-C-13924 Class 4 "Black Oxide Coating for Ferrous Metals".
- Black chromium plating: MIL-C-14538, "Chromium Black, Electroplated".

Additional procedures in the program included:

- Activation by glass-bead blasting (control).
- Passivation with a solution conforming to QQ-P-35 Type 3, "Passivation Treatments for Corrosion Resisting Steel" (control).
- Zinc phosphating according to MIL-P-16232, "Phosphate Coatings for Ferrous Metals".
- Passivation after an alkaline-oxidizing blackening treatment.
- Anodic passivation of an alkaline-oxidized specimen at 8-10 amps per square foot with a 22% (volume) nitric acid-2% (weight) sodium dichromate solution.

Prepared specimens were exposed to the 5% salt-spray test (ASTM-B117) and to a potentiostatic and potentiodynamic corrosion test. Activated (glass-bead blasted) and passivated specimens were used as controls. The specimens were potentiostatically tested for one hour at 0.1 volt versus the standard calomel electrode (SCE) in a 1.0% sodium chloride solution. The test apparatus is shown in Figure 1. Specimens for the potentiodynamic test were placed in the 0.25% sodium chloride solution after nitrogen had been bubbled through the solution for one-half hour. Specimens were anodically polarized in 10-millivolt increments every two minutes. The resulting current was recorded after one and one-half minutes. The solution temperature was maintained at 75 ± 2°F.

TABLE 2

Precipitation-Hardening Treatments

Alloy	Conditioning	Precipitation Hardening	Final Designation
17-4			17-4
17-4		1 hour @900°F	H900
17-4		4 hours @1025°F	H1025
17-4		4 hours @1150°F	H1150
17-7			17-7
17-7	90 min @1400°F and 1 hour @60°F	90 min @1050°F	TH1050
17-7	10 min @1750°F and 8 hours @-100°F	1 hour @950°F	RH950
14-8			14-8
14-8	1 hour @1700°F and 8 hours @-100°F	1 hour @950°F	SRH950
14-8	1 hour @1700°F and 8 hours @-100°F	1 hour @1050°F	SRH1050
AM350			AM350
AM350	10 min @1700°F and 8 hours @100°F	1 hour @950°F	AM350H
17-4 (Casting)	1 hour @1500°F	3 hours @1000°F	17-4 (Casting)

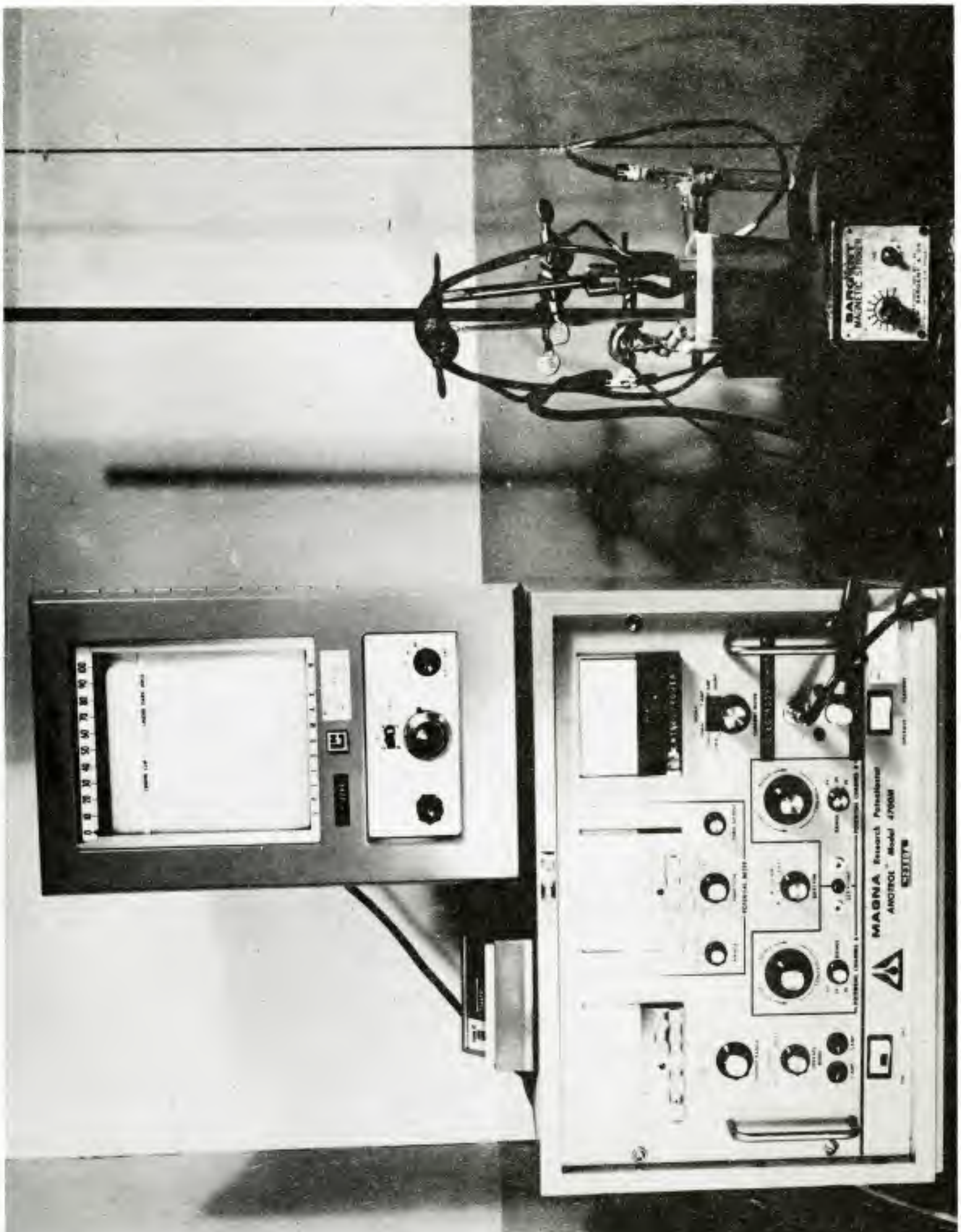

Figure 1. Equipment Used to Conduct Electrochemical Corrosion Tests

RESULTS AND DISCUSSION

Salt-Spray Test Results.

The results of the 96-hour exposure to the salt-spray test are summarized in Table 3. All control specimens exhibited minimal corrosion. Molten dichromate and alkaline-oxidation treated specimens had corroded to various degrees, dependent upon the alloy, thermal treatment or blackening process employed. In general, the molten dichromate (Class 3) coating had greatly reduced the inherent corrosion resistance of the 17-4, 17-7, and 14-8Mo steels. Alkaline-oxidation (Class 4) coatings on the 17-4PH and the 17-7PH specimens exhibited poor corrosion resistance. The solution-annealed 17-7, and the 14-8Mo and AM350 specimens with a Class 4 coating "passed" the 96-hour salt-spray exposure.

Annealed and precipitation-hardened specimens given black chromium or zinc phosphate coatings exhibited no corrosion after 96 hours of salt-spray exposure (MIL-C-13924 Class 4 requirement). These two coatings provided the best protection against salt-spray corrosion for all annealed and precipitation hardened specimens. Sections of 17-4 investment castings given black chromium or zinc phosphate coatings exhibited minimal corrosion after 96 hours of salt spray exposure.

Specimens which were passivated after blackening did not show any improvement over the blackened-only specimens in the salt-spray test. Anodic passivation treatment of blackened specimens at 8-10 amps per square foot in a nitric acid-sodium dichromate solution attacked and stripped the black coating.

Potentiostatic Tests.

Potentiostatic testing was conducted at 0.1 volt versus standard calomel electrode for one hour, and the current at the end of the test period was recorded. The current readings for all the specimens evaluated are summarized in Table 4. The condition observed is that the higher the final current, the poorer the corrosion resistance (reference Table 3). Good correlation was observed between these test results and the 5% salt-spray test results for molten dichromate and alkaline-oxidizing blackened specimens (Figures 2 and 3). Variations from the 5% salt-spray test and the potentiostatic test results were observed with the black chromium plate and the zinc phosphate coated specimens.

Potentiodynamic Tests.

Potentiodynamic tests were conducted to determine the critical pitting potential of the blackened and control specimens. Generally, almost all literature lists critical pitting potential data of stainless steels in sulfuric acid environments. However, such data are

TABLE 3

Salt-Spray Test Results

ALLOY-THERMAL TREATMENT	BLACK CHROMIUM	MIL-C-13924 CLASS 3	MIL-C-13924 CLASS 4	PHOSPHATED	PASSIVATED (CONTROL)	ACTIVATED (CONTROL)
17-4 Annealed	Passed*	Failed	Failed	Passed	Passed	Passed
17-4 H900	Passed	Failed	Failed	Passed	Passed	Passed
17-4, H1025	Passed	Failed	Failed	Passed	Passed	Passed
17-4 H1150	Passed	Failed	Failed	Passed	Passed	Passed
17-7 Annealed	Passed	Failed	Passed	Passed	Passed	Passed
17-7 TH1050	Passed	Failed	Failed	Passed	Passed	Passed
17-7 RH950	Passed	Failed	Failed	Passed	Passed	Passed
14-8 Annealed	Passed	Failed	Passed	Passed	Passed	Passed
14-8 SRH1050	Passed	Failed	Passed	Passed	Passed	Passed
14-8 SRH950	Passed	Failed	Passed	Passed	Passed	Passed
AM350 Annealed	Passed	Passed	Passed	Passed	Passed	Passed
AM350H	Passed	Passed	Passed	Passed	Passed	Passed
17-4 Casting	Passed	Failed	Failed	Passed	Passed	Passed

* Observations made after exposure to 96 hours in the 5% salt-spray test

TABLE 4

Milliamp Readings

ALLOY-THERMAL TREATMENT \ SURFACE TREATMENT	BLACK CHROMIUM	MIL-C-13924 CLASS 3	MIL-C-13924 CLASS 4	PHOSPHATED	PASSIVATED (CONTROL)	ACTIVATED (CONTROL)
17-4 Annealed	26*	61	15	500	0.36	13
17-4 H900	38	45	2.5	---	9.0	11
17-4 H1025	230	87	5.5	---	12.0	10
17-4 H1150	145	120	14	430	7.4	13
17-7 Annealed	21	230	6.5	25	0.00	11
17-7 TH1050	150	59	14	---	4.6	12
17-7 RH950	350	240	350	80	50	220
14-8 Annealed	0.35	17	0.006	0.24	0.24	1.0
14-8 SRH1050	0.32	2.8	0.37	0.18	0.27	6.0
14-8 SRH950	0.09	3.5	0.015	---	0.002	1.7
AM350 Annealed	0.02	11	0.03	---	0.00	10
AM350 H	0.23	15	1.2	---	0.06	14

* Final milliamp reading after one hour in the potentiostatic test.

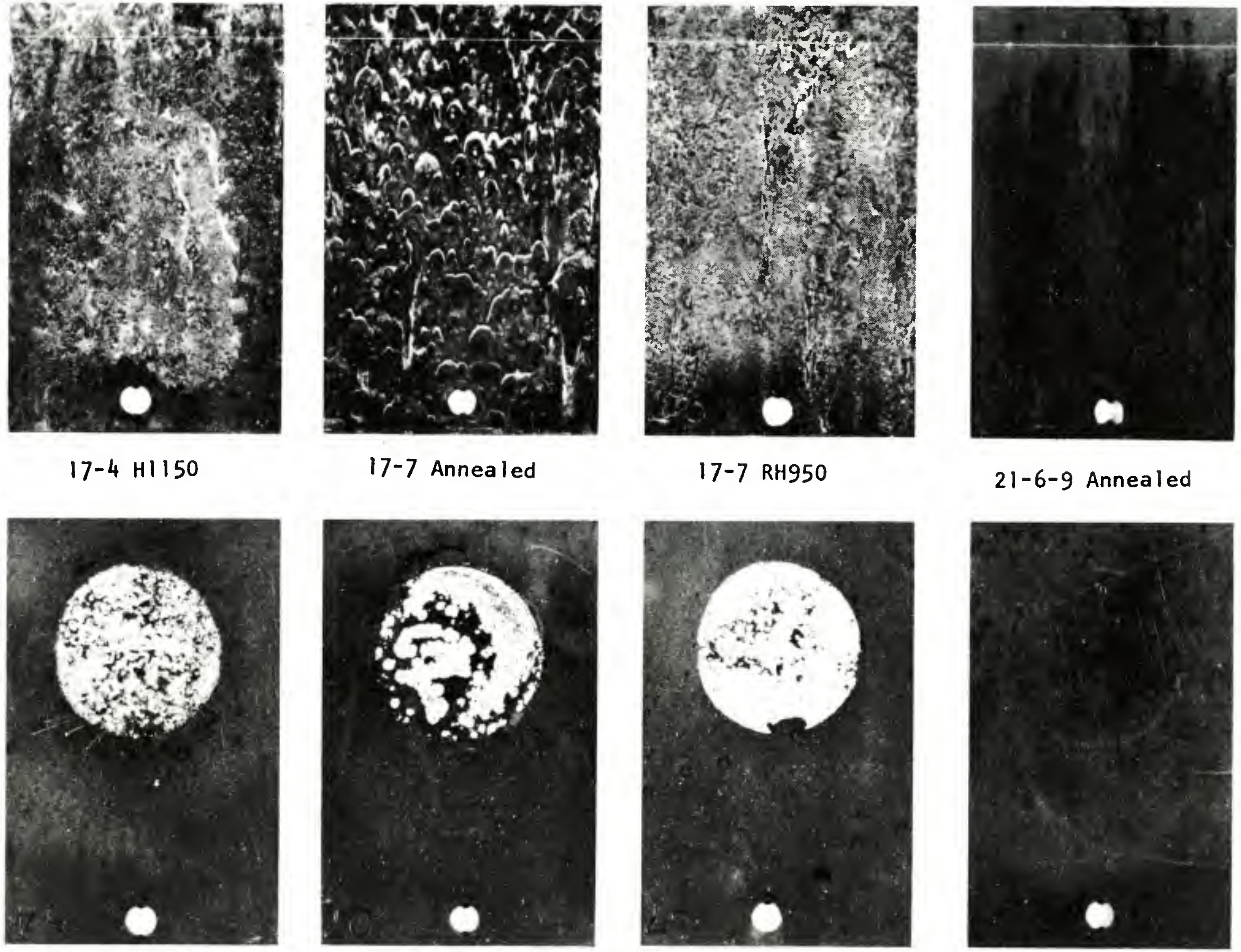

Figure 2. Comparison of Potentiostatic Testing (Bottom Row) with 5% Salt Spray Testing (Top Row) on Class 3 (Molten Dichromate) Blackened Specimens

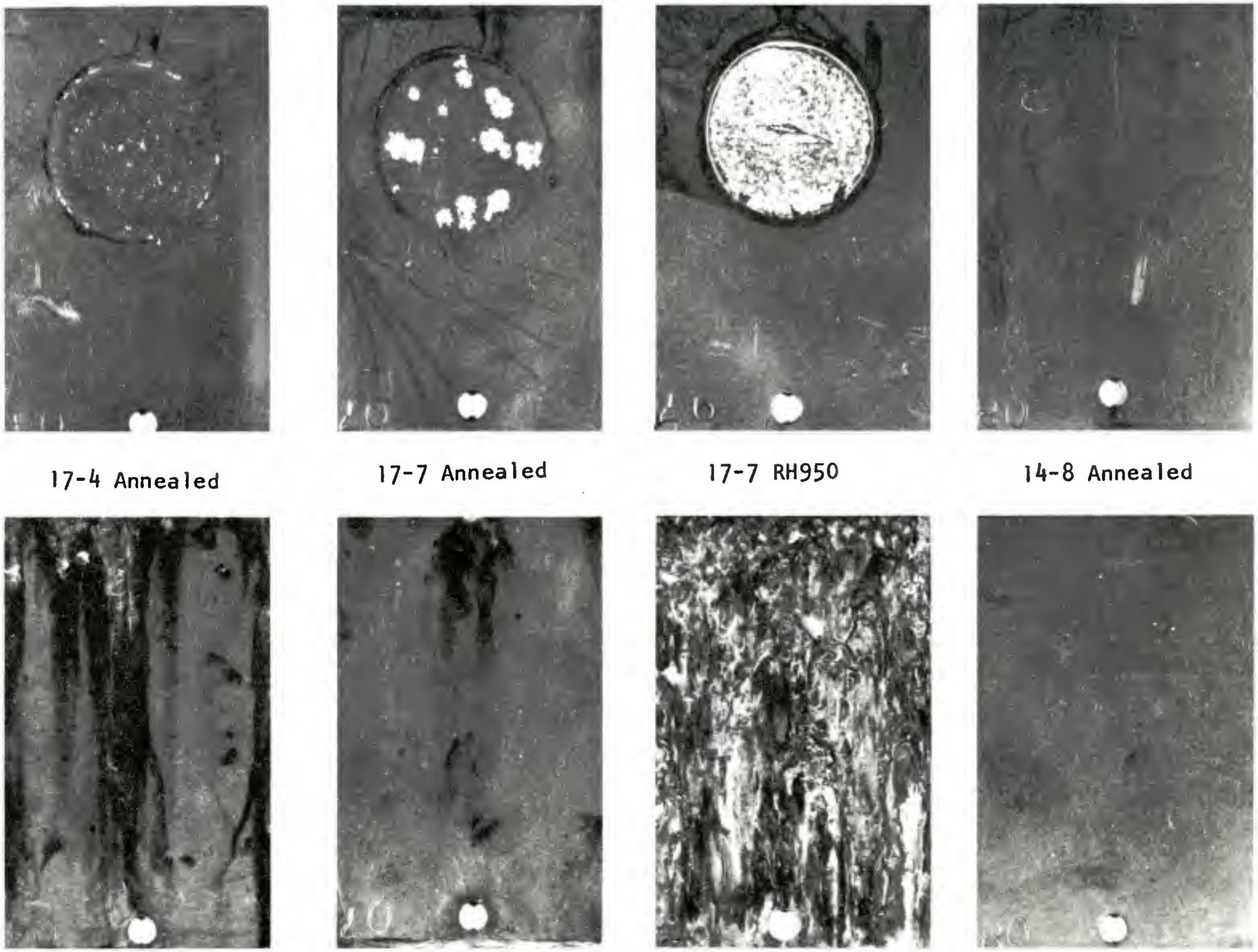

Figure 3. Comparison of Potentiostatic Testing (Top Row) with 5% Salt Spray Testing (Bottom Row) on Class 4 (Alkaline-Oxidizing) Blackened Specimens

not readily applicable to service environments to which Army weapon components have been exposed. Also, the susceptibility of the black coatings to sulfuric acid attack further complicates the situation. Corrosion of weapon components is generally observed in humid or marine environments or as a result of human perspiration residues. For these reasons, electrochemical testing was modified to use sodium chloride solutions rather than sulfuric acid solutions. The critical pitting potentials for the specimens evaluated in 0.25% sodium chloride solutions are summarized in Table 5. In comparison of these data with those of Table 3, the observation is that the higher the critical pitting potential, the better the corrosion resistance. Potentiodynamic test data are in good agreement with the potentiostatic test data with the exception of the TH1050 and RH950 specimens.

Another variable was that of the difference between the critical pitting potential of the 17-4 PH wrought specimens and the specimens obtained from a 17-4 PH investment casting. The specimens obtained from the investment casting were surface-ground before blasting and/or coating to completely eliminate the presence of Cr-depleted surface layers and heat-treat scale. Even though these precautions were taken, the critical pitting potentials of the casting specimens were considerably lower than those obtained from the wrought specimens. This indicates that the cast structures are more susceptible to corrosion than the wrought forms are. This situation may be due to structural and chemical inhomogeneities as a result of the solidification process.

Electrochemical corrosion test results, in general, are in good agreement with the salt-spray test results with the exception of the black chromium plated and the zinc phosphate coated specimens. The reason for this is that the black chromium plating may provide anodic protection to the stainless steel substrate by which the salt spray protection is increased. Zinc phosphate coating on stainless steel provides a condition in which surface corrosion is under cathodic control. Phosphate coatings form on cathodic sites thus reducing the cathode areas on the stainless surface. This condition provides a surface with a large anode-small cathode area relationship which drastically decreases anodic corrosion. Since anode and cathode areas cannot be distinguished with electrochemical solutions, the application of anodic current begins to immediately corrode anode areas disregarding protective coatings and anode-cathode relationships.

CONCLUSIONS

1. Black chromium plating improves the corrosion resistance of precipitation-hardened stainless steels.

TABLE 5

Critical Pitting Potentials (Volts vs. SCE)

ALLOY \ SURFACE TREATMENT	BLACK CHROMIUM	CLASS 3	CLASS 4	PASSIVATED (CONTROL)	ACTIVATED (CONTROL)
17-4 Annealed	0.17*	-0.22	0.17	0.52	0.15
17-4 H1100	0.20	-0.25	0.20	0.50	0.16
17-4 H900	0.21	-0.23	0.21	0.56	0.12
17-4 Casting	0.14	----	0.11	----	0.05
17-7 Annealed	0.25	-0.20	0.24	0.44	0.25
17-7 TH1050	0.08	-0.24	0.01	0.30	0.08
17-7 RH950	0.17	-0.23	0.20	0.33	0.14
14-8 Annealed	0.35	-0.13	0.28	0.64	0.28
14-8 SRH1050	0.25	-0.09	0.27	0.66	0.31
14-8 SRH950	0.29	-0.13	0.36	0.63	0.26
AM350 Annealed	0.39	-0.10	0.40	0.99	0.35
AM350 H	0.28	-0.11	0.33	0.62	0.23

17-4 Casting (Zinc Phosphate Coated) -0.09

* Critical pitting potentials obtained by potentiodynamic testing.

2. The blackening of PH stainless steels with alkaline-oxidizing treatments slightly reduces corrosion resistance.

3. Zinc phosphate coatings improve the corrosion resistance of precipitation-hardened stainless steel, but form a gray-colored surface.

4. Molten dichromate coatings significantly reduce the inherent corrosion resistance of precipitation-hardened stainless steels.

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Report EN-79-01, Jun 79, 18 p. incl. illus. tables, (PRON A1-9-23016-01-M1-M1, AMS Code 4932.06.6796). Unclassified report.

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